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2-Hydroxy-*N*′-methylbenzohydrazide

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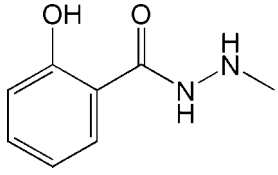
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.002 Å; R factor = 0.048; wR factor = 0.129; data-to-parameter ratio = 15.4.

In the title molecule, $C_8H_{10}N_2O_2$, there is an intramolecular hydrogen bond involving the hydroxy group and the O atom of the carbonyl group. The dihedral angle between the benzene ring and the amide fragment is 87.16 (10)°. The C—N—N—C torsion angle is 88.87 (18)°. In the crystal, N—H···N and N—H···O hydrogen bonds connect molecules into chains along [100]. In addition, there is a weak C—H···π interaction.

Related literature

For applications of related materials, see: Zhang *et al.* (2012); Jin *et al.* (2011). For the preparation of the title compound, see: Li *et al.* (2001). For a related structure, see: Jin (2007).



Experimental

Crystal data

$C_8H_{10}N_2O_2$
M_r = 166.18
Monoclinic, $P2_1/c$
a = 7.4863 (10) Å
b = 14.706 (2) Å
c = 7.7232 (11) Å
β = 96.898 (2)°
V = 844.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.981
6478 measured reflections
1837 independent reflections
1381 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.129
S = 1.03
1837 reflections
119 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

Cg is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*B*···O2	0.91 (2)	1.72 (2)	2.5535 (15)	150 (2)
N1—H1*A*···N2^{i}	0.859 (19)	2.16 (2)	2.9415 (18)	151.9 (15)
N1—H1*A*···N1^{i}	0.859 (19)	2.619 (18)	3.1403 (18)	120.4 (13)
N2—H2*A*···O2^{ii}	0.884 (18)	2.253 (17)	2.9866 (16)	140.3 (14)
C4—H4···*Cg*^{iii}	0.93	2.83	3.6713 (13)	152

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+2, -y+1, -z$; (iii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5501).

References

Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jin, L.-F. (2007). *Acta Cryst.* E**63**, o3465.
Jin, C. Z., Yang, Y., Jin, L. F., Wu, L. M. & Zhang, J. (2011). *J. Coord. Chem.* **64**, 4098–4107.
Li, Z. G., Wang, Q. M. & Huang, J. M. (2001). In *Preparation of Organic Intermediate Compounds*. Beijing: Chemical Industry Publishing House.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhang, X. W., Wu, L. M., Zhang, J. & Jin, L. F. (2012). *Synth. React. Inorg. Met. Org. Nano-Met. Chem.* **42**, 87–91.

supplementary materials

Acta Cryst. (2012). E**68**, o2686 [doi:10.1107/S160053681203468X]

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Comment

Materials such as the title compound and the recently determined crystal structure of 4-(5-bromo-2-hydroxybenzoyl)thiosemicarbazide (Jin, 2007) are potentially important ligands and intermediates (Zhang *et al.*, 2012; Jin *et al.*, 2011). Part of our studies is to find new methods to synthesize derivatives of saylicylic acid and study their structures and activities. In this paper, the X-ray crystal structure determination of the title compound (I) is reported.

The molecular structure of the title compound is shown in Fig. 1. The geometric parameters of (I) agree with those related in the structure published by Jin (2007). The atoms C1—C7/O1/O2 are essentially co-planar with an r.m.s deviation of 0.016Å. There is intramolecular hydrogen bond involving the hydroxy group and the O atom of the carbonyl group. In the crystal, N—H···N and N—H···O hydrogen bonds connect molecules into one-dimensional chains along [100] (see Table 1 and Fig. 2). In addition, there is a weak intermolecular C—H···π interaction. There are some intermolecular contacts which are shorter than the sums of the van der Waals radii of the atoms involved i.e. N1···N2^{iv} [2.9415 (18) Å], O2···N2^{v} [2.9866 (17) Å] and C6···C6^{vi} [3.346 (2) Å] [symmetry code (iv): 1 - *x*,-*y*,1 - *z*; (v): -*x*,-*y*,1 - *z*; (vi): 1 - *x*,-*y*,-*z*].

Experimental

Methyl salicylate (15.2 g, 0.10 mol) and methylhydrazine in aqueous solution (23.0 g, 0.20 mol) were mixed at 273 K and stirred for 1 h. The reaction mixture was slowly warmed to 338 K and refluxed for a further 12 h. After the resulting mixture was concentrated under reduced pressure, the residue was adjusted to pH 8 with acetic acid. After staying for 1 h in a refrigerator, the resulting precipitate was filtered and rinsed with ethyl ether. A white solid formed was recrystallized to give 7.0 g (42% yield) of *N*-methyl-salicylhydrazide. A block-like crystal suitable for X-ray analysis was grown from a solution of the title compound in methanol at room temperature by slow evaporation.

Refinement

The hydroxy H atom was located in a difference Fourier map and refined isotropically [O—H = 0.91 (2) Å] with U_{iso}(H) = 1.5U_{eq}(O). The H atoms bonded to N atoms were located in a difference Fourier map and refined isotropically (N—H = 0.859 (19) and 0.884 (18) Å) with U_{iso}(H) = 1.2U_{eq}(N). All other H atoms were included in a riding-model approximation, with C—H distances of 0.93 (aromatic H atoms) and 0.96 Å (methyl atoms). The isotropic displacement parameters were set to 1.2U_{eq}(C) for the aromatic H atoms and to 1.5U_{eq}(C) for the methyl H atoms.

Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

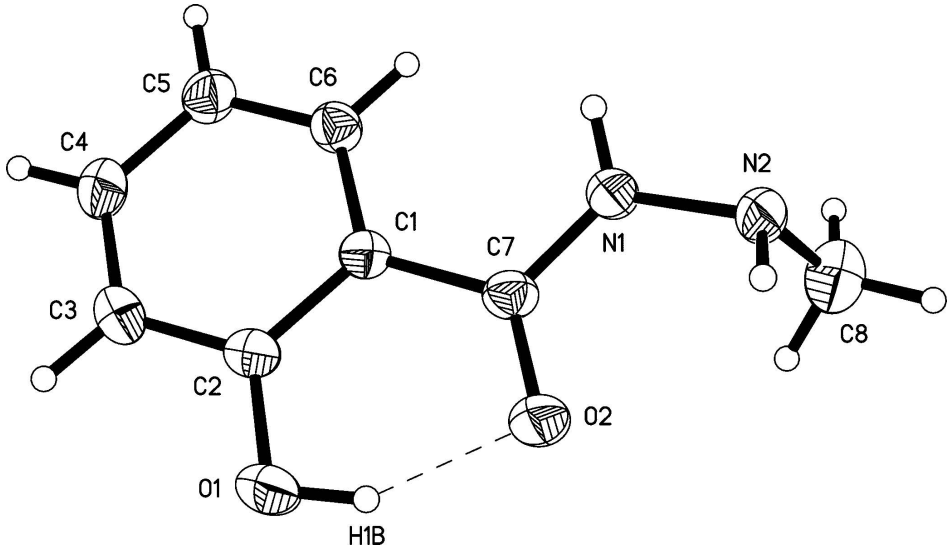

Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids. The dashed lines indicates a hydrogen bond.

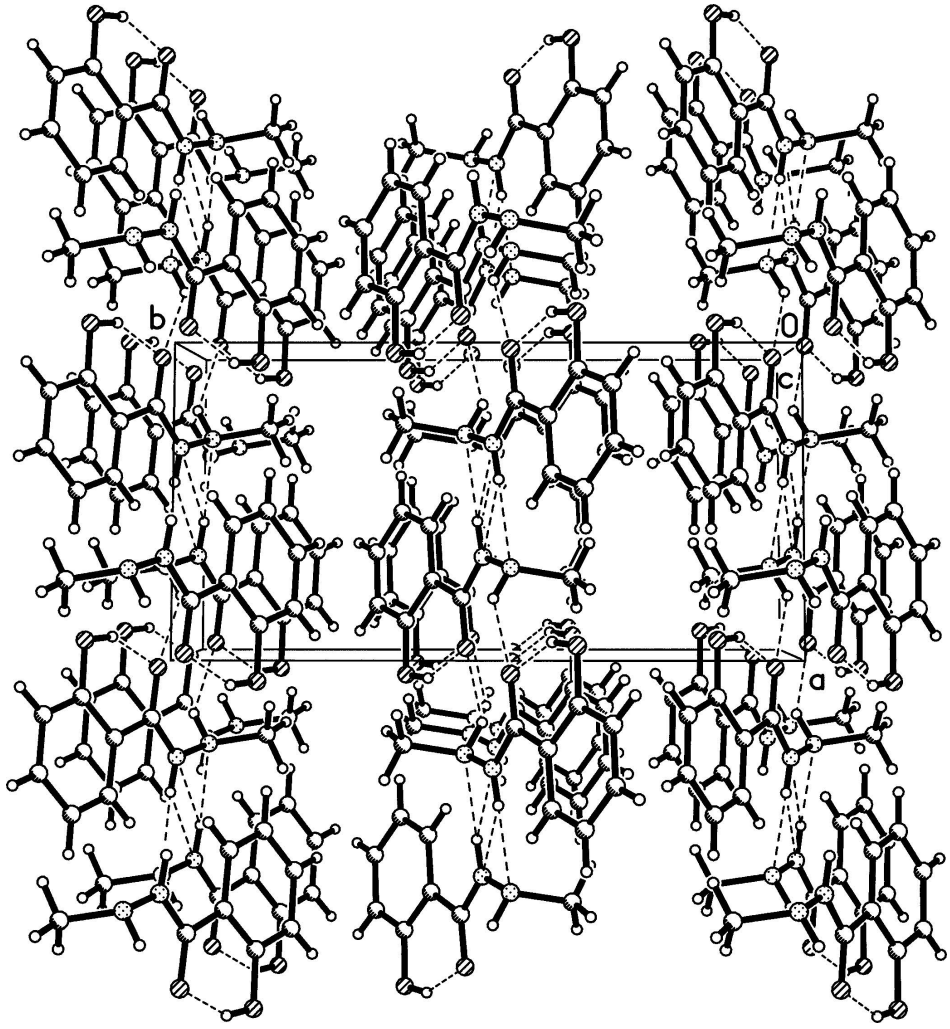

Figure 2

Packing diagram for (I). The hydrogen bonds are indicated by dashed lines.

2-Hydroxy-*N*′-methylbenzohydrazide

Crystal data

$C_8H_{10}N_2O_2$
M_r = 166.18
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.4863 (10) Å
b = 14.706 (2) Å
c = 7.7232 (11) Å
β = 96.898 (2)°
V = 844.1 (2) Å^3
Z = 4
$F(000)$ = 352
D_x = 1.308 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1880 reflections
θ = 2.7–26.5°
μ = 0.10 mm^{-1}
T = 294 K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.981

6478 measured reflections
1837 independent reflections
1381 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 27.0°, θ_{min} = 2.7°
h = −7→9
k = −16→18
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.129
S = 1.03
1837 reflections
119 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.75411 (17)	0.59342 (9)	0.39608 (15)	0.0389 (3)
C2	0.88807 (19)	0.63880 (10)	0.50734 (17)	0.0467 (4)
C3	0.8400 (2)	0.68846 (12)	0.6475 (2)	0.0593 (4)
H3	0.9278	0.7199	0.7192	0.071*
C4	0.6643 (2)	0.69159 (11)	0.68107 (19)	0.0576 (4)
H4	0.6344	0.7240	0.7769	0.069*
C5	0.5313 (2)	0.64697 (10)	0.57369 (18)	0.0496 (4)
H5	0.4122	0.6494	0.5965	0.060*
C6	0.57723 (19)	0.59916 (9)	0.43321 (17)	0.0432 (4)
H6	0.4874	0.5697	0.3605	0.052*
C7	0.80781 (18)	0.54385 (10)	0.24308 (16)	0.0423 (4)
C8	0.7418 (3)	0.36724 (13)	0.0064 (2)	0.0769 (6)
H8A	0.8366	0.3593	0.1005	0.115*
H8B	0.7758	0.3395	−0.0973	0.115*
H8C	0.6338	0.3392	0.0363	0.115*
N1	0.67678 (17)	0.50872 (10)	0.13066 (14)	0.0514 (4)

H1A	0.565 (3)	0.5176 (12)	0.139 (2)	0.062*
N2	0.71005 (17)	0.46382 (10)	−0.02434 (14)	0.0498 (4)
H2A	0.805 (2)	0.4910 (11)	−0.058 (2)	0.060*
O1	1.06320 (14)	0.63710 (9)	0.48180 (15)	0.0688 (4)
H1B	1.071 (3)	0.6000 (15)	0.388 (3)	0.103*
O2	0.96759 (14)	0.53627 (8)	0.21771 (13)	0.0597 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0367 (8)	0.0413 (8)	0.0386 (7)	0.0003 (6)	0.0045 (5)	0.0015 (5)
C2	0.0368 (8)	0.0526 (9)	0.0497 (7)	0.0011 (6)	0.0014 (6)	−0.0013 (6)
C3	0.0537 (10)	0.0643 (11)	0.0576 (9)	−0.0045 (8)	−0.0034 (7)	−0.0194 (7)
C4	0.0604 (11)	0.0587 (10)	0.0547 (8)	0.0022 (8)	0.0112 (7)	−0.0166 (7)
C5	0.0436 (9)	0.0534 (9)	0.0540 (8)	−0.0020 (7)	0.0154 (6)	−0.0050 (7)
C6	0.0397 (8)	0.0466 (8)	0.0438 (7)	−0.0051 (6)	0.0064 (5)	−0.0015 (6)
C7	0.0339 (8)	0.0515 (9)	0.0422 (7)	−0.0005 (6)	0.0069 (5)	0.0013 (6)
C8	0.0874 (15)	0.0714 (14)	0.0732 (11)	0.0043 (10)	0.0149 (9)	−0.0133 (9)
N1	0.0346 (7)	0.0761 (9)	0.0442 (6)	0.0007 (6)	0.0077 (5)	−0.0167 (6)
N2	0.0402 (7)	0.0668 (9)	0.0438 (6)	−0.0031 (6)	0.0113 (5)	−0.0136 (6)
O1	0.0349 (7)	0.0969 (10)	0.0734 (7)	−0.0048 (6)	0.0013 (5)	−0.0247 (7)
O2	0.0352 (6)	0.0868 (9)	0.0586 (6)	−0.0012 (5)	0.0113 (4)	−0.0172 (5)

Geometric parameters (Å, º)

C1—C6	1.3907 (19)	C6—H6	0.9300
C1—C2	1.4081 (18)	C7—O2	1.2401 (16)
C1—C7	1.4844 (18)	C7—N1	1.3336 (18)
C2—O1	1.3493 (17)	C8—N2	1.455 (2)
C2—C3	1.388 (2)	C8—H8A	0.9600
C3—C4	1.372 (2)	C8—H8B	0.9600
C3—H3	0.9300	C8—H8C	0.9600
C4—C5	1.383 (2)	N1—N2	1.4152 (16)
C4—H4	0.9300	N1—H1A	0.859 (19)
C5—C6	1.3709 (19)	N2—H2A	0.884 (18)
C5—H5	0.9300	O1—H1B	0.91 (2)
C6—C1—C2	118.12 (12)	C1—C6—H6	119.0
C6—C1—C7	123.34 (11)	O2—C7—N1	120.69 (12)
C2—C1—C7	118.52 (13)	O2—C7—C1	121.90 (12)
O1—C2—C3	118.09 (13)	N1—C7—C1	117.40 (12)
O1—C2—C1	122.37 (12)	N2—C8—H8A	109.5
C3—C2—C1	119.53 (14)	N2—C8—H8B	109.5
C4—C3—C2	120.61 (13)	H8A—C8—H8B	109.5
C4—C3—H3	119.7	N2—C8—H8C	109.5
C2—C3—H3	119.7	H8A—C8—H8C	109.5
C3—C4—C5	120.57 (13)	H8B—C8—H8C	109.5
C3—C4—H4	119.7	C7—N1—N2	122.75 (12)
C5—C4—H4	119.7	C7—N1—H1A	122.9 (11)
C6—C5—C4	119.14 (14)	N2—N1—H1A	113.8 (11)

C6—C5—H5	120.4	N1—N2—C8	111.07 (12)
C4—C5—H5	120.4	N1—N2—H2A	105.5 (10)
C5—C6—C1	122.00 (13)	C8—N2—H2A	111.6 (11)
C5—C6—H6	119.0	C2—O1—H1B	106.4 (14)
C6—C1—C2—O1	179.99 (12)	C2—C1—C6—C5	−0.2 (2)
C7—C1—C2—O1	−1.5 (2)	C7—C1—C6—C5	−178.69 (12)
C6—C1—C2—C3	−1.0 (2)	C6—C1—C7—O2	−176.57 (13)
C7—C1—C2—C3	177.58 (13)	C2—C1—C7—O2	5.0 (2)
O1—C2—C3—C4	−179.12 (15)	C6—C1—C7—N1	4.7 (2)
C1—C2—C3—C4	1.8 (2)	C2—C1—C7—N1	−173.80 (13)
C2—C3—C4—C5	−1.4 (2)	O2—C7—N1—N2	−1.9 (2)
C3—C4—C5—C6	0.2 (2)	C1—C7—N1—N2	176.89 (12)
C4—C5—C6—C1	0.6 (2)	C7—N1—N2—C8	88.87 (18)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O2	0.91 (2)	1.72 (2)	2.5535 (15)	150 (2)
N1—H1*A*···N2^{i}	0.859 (19)	2.16 (2)	2.9415 (18)	151.9 (15)
N1—H1*A*···N1^{i}	0.859 (19)	2.619 (18)	3.1403 (18)	120.4 (13)
N2—H2*A*···O2^{ii}	0.884 (18)	2.253 (17)	2.9866 (16)	140.3 (14)
C4—H4···*Cg*^{iii}	0.93	2.83	3.6713 (13)	152

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+2, −*y*+1, −*z*; (iii) *x*, −*y*+3/2, *z*+1/2.